

Electrochemical and chemical investigations of the co-polymers of 3-aminobenzenesulfonic acid with aromatic amines for their application in electrochromic devices

Ritu Saharan · Amarjeet Kaur · S. K. Dhawan

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Abstract The chemical oxidative polymerization and electrochemical polymerization of 3-aminobenzenesulfonic acid with aromatic amines have been carried out in *p*-toluenesulfonic acid which acts as a supporting electrolyte as well as an external dopant. The presence of $-\text{SO}_3\text{H}$ groups in the ABSA co-monomer allows the copolymer to acquire intrinsic protonic doping ability. The electrochemically synthesized polymers and copolymers have been characterized by cyclic voltammetry for analyzing the growth of copolymers and chronoamperometry studies for their applications to the electrochromic devices. In addition to construction of electrochromic devices (ECDs), the electrochromic properties of the polymer films were further investigated. Electrochromic switching stability of the devices was estimated from switching times between their oxidized and reduced states, which indicates that the homopolymers and copolymer can be used for promising electrochromic devices. Chemically synthesized copolymers were also characterized using various techniques like Fourier transform infrared spectroscopy, ultraviolet–visible spectroscopy and electrical conductivity at room temperature.

Keywords Poly 3-aminobenzenesulfonic acid · Copolymer · Electrochemical polymerization · Electrochromic devices

1 Introduction

The study of conducting polymers like polypyrrole, polyaniline, polythiophene, etc., has been a very demanding field over recent years. The outstanding properties of these polymers, allow themselves to be used as sensors, catalysts, electrochromic materials, etc. [1–6]. However, the poor processability of these materials somehow restricts their applications. Therefore, many efforts have been carried out to make them processible by functionalizing and copolymerizing these materials [7–14]. A major consideration in the design of the copolymers is the improvement in mechanical, optical, and the electrochemical properties, with respect to those of the parent conducting polymeric materials [15–17]. In light of this, various copolymers, poly(pyrrole-*N*-methyl pyrrole), poly(pyrrole-thiophene), copoly(3-thiophenes), poly(aniline-ortho-anisidine), poly(aniline-co-ortho-ethoxyaniline), etc. have been prepared either by electrochemical or chemical polymerization techniques [18–24]. In most of the cases, the estimation of copolymerization reactivity leads to the incorrect parameters as well as a non-linear approach, for the estimation of reactivity ratios that are achieved by copolymer composition [21, 25]. Copolymers like poly(aniline-co-2-isopropylaniline) [26] materials show the switching of redox states which generates different visible region bands. Color changes in an object give visual signals that can be used to convey useful information to an observer. Moreover, by discriminating absorption or transmission of light by a material, the light energy impinging upon an observer can be modulated. In some of the conducting polymers and their copolymers [26], a chemical or physical external stimulus causes a reversible color change or variation in light transmission. The optical sensor applications of such materials are considerable. For such systems, color change

R. Saharan · A. Kaur (✉)
Department of Physics and Astrophysics, University of Delhi,
New Delhi 110007, India
e-mail: amarkaur@physics.du.ac.in

S. K. Dhawan
Polymeric and Soft Materials Section, National Physical
Laboratory, New Delhi 110012, India

phenomena are classified after the stimulus that causes the change, such as electrochromism, photochromism, thermochromism and gasochromism. An electrochromic material is one where a reversible color change takes place upon reduction (gain of electrons) or oxidation (loss of electrons), on passage of electrical current after the application of an appropriate electrode potential [27, 28]. In other words, the performance of the device is governed by how in minimum time maximum oxidation or reduction potential is achieved. Commercial applications of electrochromic materials in the devices include anti-glare car rear-view mirrors, electrochromic strips as battery state-of-charge indicators, and electrochromic sun-glasses [28–30]. Proposed applications include ‘smart windows’ (based on modulation of either the transmitted or reflected solar radiation) for their use in the cars and in buildings [10], reusable price labels, protective eyewear, controllable aircraft canopies, glare-reduction systems for offices, devices for frozen-food monitoring [16], camouflage materials, spacecraft thermal control, and controllable light-reflective or light-transmissive display devices for optical information and storage.

Electrochromic devices have been considered as the most promising application for poly(aromaticamine) poly(AA). However, problems like poor processibility and relatively slow response of poly(AA) to an electrochemical perturbation are yet to overcome entirely. Several ways such as copolymerization with aminobenzenesulfonic acid (ABSA) or substitution with 2,5 dialkoxy, have been reported in the literature for increasing the solubility of aromatic amines [7, 31]. The values of electrical conductivity of these soluble polymers are comparable to that of poly(AA). This prompted us to systematically study the electrochemical properties of poly(AA) and its copolymers for electrochromic applications. In the present investigations, we report the result of chemical synthesis of self-doped homopolymer poly(3-aminobenzenesulfonic acid-co-aromatic amine) poly(3-ABSA-co-AA), *p*-toluenesulfonic acid (PTSA) doped copolymer poly(3-ABSA-co-AA) and electrochemical synthesis of poly(AA), and their copolymer with 3-aminobenzenesulfonic acid (ABSA) in the medium of PTSA. The choice of ABSA is due to the better solubility and better processibility of the polymer in the presence of functionalized acid.

We have investigated both chemically as well as electrochemically polymerized polymers. In chemical process, there is always possibility of oligomers produced along with the main polymer, however, the bulk polymer can be produced in a single batch and the same mother polymer can be used for various investigations. The electrochemical polymerization can not be used for the bulk preparation of polymer and is suitable for obtaining films of pure polymer on electrode surface which can be used for electrochromic

devices. To optimize the composition for electrochromic applications, we have studied the electrochemical growth of poly(AA), and their copolymer poly(3-ABSA-co-AA) in PTSA medium and their characterization by cyclic voltammetry and chronoamperometry.

2 Experimental procedures

2.1 Electrochemical polymerization

3-aminobenzenesulfonic acid (3-ABSA), and aromatic amines (AA) were used as monomers. The PTSA was used as a synthesizing medium. Aqueous solutions were prepared from the double distilled water having a specific resistivity of 1.0 M Ω -cm.

The copolymerization of 3-ABSA with 0.1 M AA in 0.2 M PTSA, was carried out at 1 V by electrochemical polymerization technique. Electro-polymerization, cyclic-voltammetry and chronoamperometry investigations were carried out using Autolab30, Potentiostat/Galvanostat, Netherland. Silver chloride was used as a reference electrode, the working and the counter electrodes were platinum foils. Cyclic potential sweep technique was used and the potentials were swept continuously in the range of –0.2 to +0.80 V at various scan rates of 5, 10, 20, 50, 100 mV/s. Peak potential values of the corresponding copolymers were recorded in PTSA medium.

2.2 Chemical polymerization

The self-doped homopolymer of 3-aminobenzene sulfonic with aromatic amine of 2:1 molar ratio compositions poly(3-ABSA-co-AA) was synthesized by chemical oxidative polymerization method and in case of copolymer the molar ratio of 3-ABSA and aromatic amines was 1:1. Their copolymers were synthesized with PTSA. The concentration of PTSA was 0.2 M. Polymerization was initiated by the dropwise addition of ammonium persulfate solution (0.1 M, (NH₄)₂S₂O₈ in distilled water). The polymerization was carried out at a temperature of 0 °C for a period of 6 h. The synthesized copolymers were isolated from reaction mixture by filtration and washed with distilled water to remove oxidant and oligomers. Copolymers were collected by filtration followed by drying under vacuum oven at 50 °C.

The surface morphology of the prepared films was investigated by scanning electron microscope (Leo). The Fourier transform infrared spectra (FTIR) of various samples were recorded on Nicolet 5700 FTIR, USA, in KBr pellets in the range of 400–4,000 cm^{–1} and Ultraviolet–visible (UV–Vis) spectra were taken on UV–vis Spectrophotometer, Shimadzu UV-1601, Japan.

3 Results and discussion

By simple quantitative solubility analysis it has been observed that poly(3-ABSA-co-AA) copolymers synthesized by chemical and electrochemical polymerization have higher solubility in solvents such as DMSO (80–85% by weight) and NMP (75–80% by weight), whereas the polymer poly(AA) has the solubility around 50–60% in these solvents. The solubility may vary with the solvents, as reported by other authors [32, 33]. The higher solubility of the copolymers relative to that of aromatic amine may be due to the ability of the solvent to act as hydrogen-bond acceptors and interact with the amine group, replacing the interchain hydrogen bonding between the amine and imine groups of the adjacent chains (acceptors), to aid in solubilizing the polymer. The difference in the solubilities among the copolymers may arise from the differences in the chain lengths and/or the number of $-\text{SO}_3$ groups incorporated into the chain. The chemically synthesized polymer is more soluble as the oligomers generated during the synthesis lead to more soluble products as compared to that prepared by electrochemical technique.

The FTIR spectra of self-doped homopolymer poly(3-ABSA-co-AA) and PTSA-doped copolymer poly(3-ABSA-co-AA) are given in Fig. 1. The FTIR spectra of self-doped poly(3-ABSA-co-AA) display intense bands at 1,574 and 1,497 cm^{-1} , which are assigned to the ring stretching vibrations of the quinoid and benzenoid ring, respectively. In case of PTSA-doped copolymer these bands shifted to lower wave numbers 1,560 and 1,482 cm^{-1} , respectively. The other bands which were present on self-doped poly(3-ABSA-co-AA) copolymer

were observed at around 1,032, 1,159, and 1,243 cm^{-1} . These bands can be assigned to the symmetrical stretching of $-\text{SO}_3$ group, asymmetrical stretching of $-\text{SO}_3$ group and C–N stretching of aromatic amine, respectively. Also, for the case of PTSA-doped copolymer these bands are shifted at lower wave numbers 1,028, 1,116, and 1,240 cm^{-1} , respectively. A band at 993 cm^{-1} is assignable to the ring-breathing mode of the quinoid group, which becomes active on rotation. The NH_2 wagging of the protonated group is found at 701 cm^{-1} . For PTSA-doped copolymer these bands shifted at the wave numbers 1,004 and 704 cm^{-1} , respectively [41]. Similar results were obtained for electrochemically prepared copolymer. Almost all the prominent bands observed in the chemically prepared polymers/copolymers are present in the electrochemically prepared polymer/copolymers. As a representative result, the FTIR spectrum of electrochemically prepared PTSA-doped poly(3-ABSA-co-AA) is given in Fig. 1. However, some small shifts in the band positions have been observed which may be due to the fact that the electrochemically prepared polymers are more pure due to the absence of oligomers and other impurities.

Figure 2 elucidates the UV–Vis spectra of self-doped homopolymer of poly(3-ABSA-co-AA), PTSA-doped poly(3-ABSA-co-AA) and its electrochemical counter part, taken in NMP solutions. As a representative result, the band energy spectra of the homopolymer and copolymer are shown in the inset of Fig. 2. Two major bands located at 278 and 628 nm with a mark shoulder at ~ 331 nm have been observed for poly(3-ABSA-co-AA) homopolymer with corresponding band energies 1.70 and 3.81 eV, respectively. However, the PTSA-doped poly(3-ABSA-

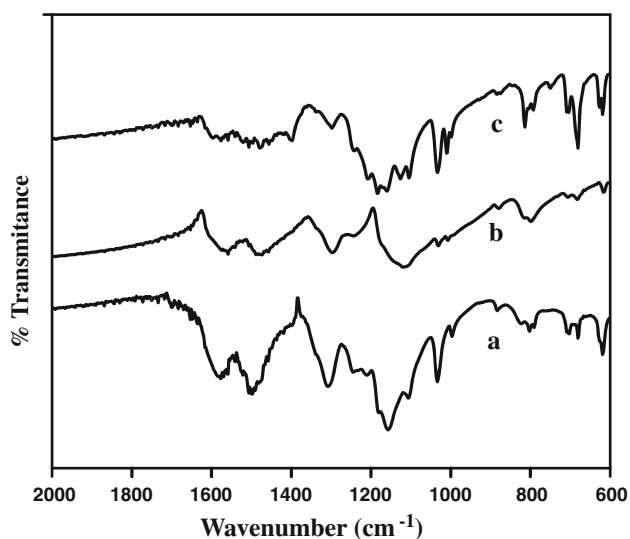


Fig. 1 FTIR spectra of **a** self-doped poly(3-ABSA-co-AA), **b** PTSA-doped poly(3-ABSA-co-AA) and **c** electrochemically prepared PTSA-doped poly(3-ABSA-co-AA)

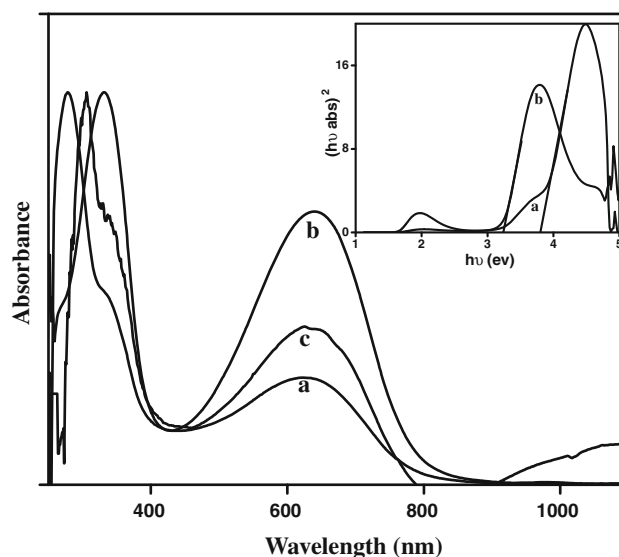


Fig. 2 UV–vis spectra of self-doped **a** poly(3-ABSA-co-AA), **b** PTSA-doped poly(3-ABSA-co-AA) prepared by chemical oxidation and **c** electrochemically prepared PTSA-doped poly(3-ABSA-co-AA)

co-AA) has only two peaks located at 331 and 642 nm with enlarge intensity (Fig. 2b) and lower band energies 1.66 and 3.25 eV. The bands in both the samples around 278 and 331 nm are assigned to the $\pi-\pi^*$ transitions in the benzenoid rings while the second band at 628 nm (3-ABSA-co-AA) and 642 nm (PTSA-doped 3-ABSA-co-AA) is attributed to the transitions $n-\pi^*$ from the highest occupied molecular orbital of the benzenoid ring (lone, non-bonding nitrogen pair) to the lowest unoccupied molecular orbital (π^*) of the quinoid ring [41]. In case of copolymer the exciton band near 642 nm undergoes a bathochromic shift when the PTSA is an external dopant. It suggests an increase in the extent of conjugation and a decrease in the bandgap. The band at 278 nm in the self-doped homopolymer shifts to 331 nm in the spectra of the PTSA-doped copolymers, indicating once again an increase in the extent of conjugation, favoring chelate formation, as observed for sulfonated polyaniline. The room temperature conductivity of PTSA-doped poly(3-ABSA-co-AA) ($4.34 \times 10^{-2} \text{ S cm}^{-1}$) is better than that of self-doped poly(3-ABSA-co-AA) ($7.734 \times 10^{-4} \text{ S cm}^{-1}$).

λ_{max} of electrochemically prepared poly(3-ABSA-co-AA) lies at higher wavelength (645 nm for $n-\pi^*$ transition and 346 nm for $\pi-\pi^*$ transition). The corresponding band gap decreases (1.56, 3.17 eV) in the electrochemically prepared poly(3-ABSA-co-AA) and has conductivity higher by one order as compared with chemically synthesized poly(AA) and poly(3-ABSA-co-AA). The higher conductivity might be due to higher conjugation in the electrochemically prepared poly(AA) and poly(3-ABSA-co-AA).

The polymer films were grown electrochemically (Fig. 3) on the platinum electrode (working) in aqueous PTSA medium at room temperature using cyclic potential sweep techniques. Peak potential values of the

corresponding polymer and the copolymer were recorded in PTSA medium. The electrochemical copolymerization growth of poly(3-ABSA-co-AA), the electrochemical polymerization growth of poly(AA), and the current values of each oxidation and reduction peaks are greater than that of a previous cycle which indicates the built up of an electroactive polymeric material on the platinum electrode. The surface of the working electrode undergoes further reaction to give oligomer species that are more readily oxidized and finally after a few coupling, the intensity of peak-I increases showing the growth of the polymer film, hence the increase of film thickness at the platinum electrode surface. On comparing the electrochemical growth behavior of poly(AA) and its copolymer with poly(3-ABSA-co-AA), it has been observed that the cyclic voltammogram (CV) of aromatic amine growth in PTSA medium differs from the growth behavior observed in their copolymers. This further confirms that the growth behavior of poly(AA) and its copolymers by electrochemical technique depends upon the oxidation potential of co-monomers. It is well known that the intensity of peak-I is a measure of the polymer formation rate [36, 38]. Figure 3a shows the different values of anodic currents corresponding to the peak-I (at 20 mV) against the number of cycles for the substrates indicate different values of conductivities. It is obvious that the current density at the substrate surface is a function of the substrate conductivity and it is used to oxidize the monomer as well as the intermediate species generated during the polymerization process. The intermediate species consist of the soluble and insoluble products formed during the polymerization process. The soluble species diffuse into the electrolyte whereas the insoluble species adhere to the electrode surface and further facilitate the polymerization process. It has been already seen [34, 35] that the efficiency of the polymerization process depends on the substrate conductivity and it increases with increasing the substrate conductivity. Therefore, this observation signifies that the generation of soluble species should decrease with increasing the substrate conductivity [36].

Cyclic voltammetry is a highly successful method that qualitatively reveals the reversibility of electron transfer during electrochemical polymerization and also examines the electrical activity of copolymer films by monitoring the oxidation and reduction in the form of a current–potential diagram (cyclic voltammogram). To investigate the electrochemical polymerization behavior of the poly(AA) and the effect on the copolymerization process [37–39], the CV for aromatic amine and poly(3-ABSA-co-AA) films was obtained by potential sweeping in a blank 0.2 M PTSA medium. Figure 4a shows the first peak value at 0.166 V for poly(AA) and Fig. 4b shows two main redox couples at +0.175 V (peak-I) and +0.669 V (peak-II) for poly(3-ABSA-co-AA). The electrochemical activities of poly(AA)

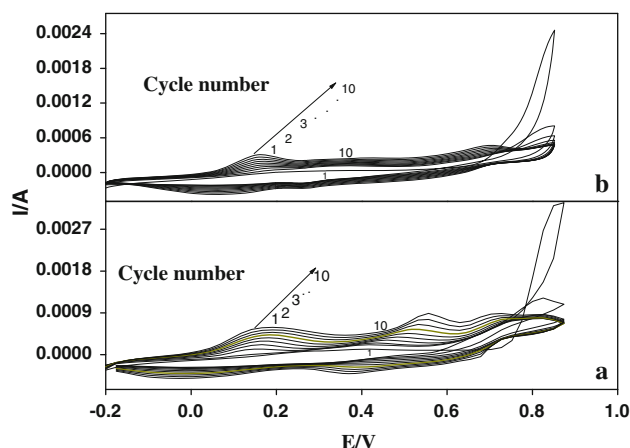


Fig. 3 Electrochemical growth in PTSA medium **a** poly(AA) and **b** poly(3-ABSA-co-AA)

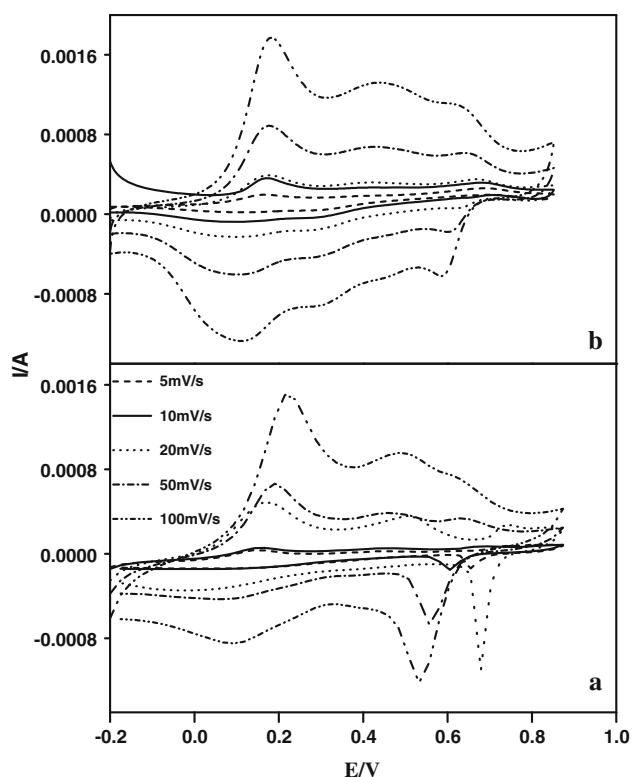


Fig. 4 Cyclic Voltammetry of **a** poly(AA) and **b** poly(3-ABSA-co-AA)

and its copolymers describe the configuration of radical cations near peak-I, which are consequently oxidized in the imines near peak-II. The first peak is assigned to the transformations of leucoemeraldine to emeraldine salt, and the second peak is assigned to the transition from emeraldine salt to the pernigraniline state. The third peak in middle, basically appropriate to the quinone/hydroquinone couple, has support from studies on chemically modified electrodes which report the strong adsorption of the quinone/hydroquinone couple on the electrode surface [40, 41].

The CVs of poly(AA) and poly(3-ABSA-co-AA) at various scan rates have been shown in Fig. 4a, b. The anodic peak current of different copolymers with respect to first peak at different scan rate differs from one another. Figure 4a shows that the anodic peak current with respect to first peak-I increases with increase in the scan rate. It can also be seen from Fig. 4b that the oxidation peak potential of first peak shifts to more positive with increasing scan rate; this indicates that there exists a kinetics limitation in the reaction between the redox behavior of copolymer films [41].

In chemically synthesized polyaromatic amine and copolymer, CV (Fig. 5) was recorded in non-aqueous medium containing tetra-*n* butylammonium tetrafluoroborate (TBAF₄). The polymer was first dissolved in non-aqueous solvent. CV data show broad peaks at 0.43 V

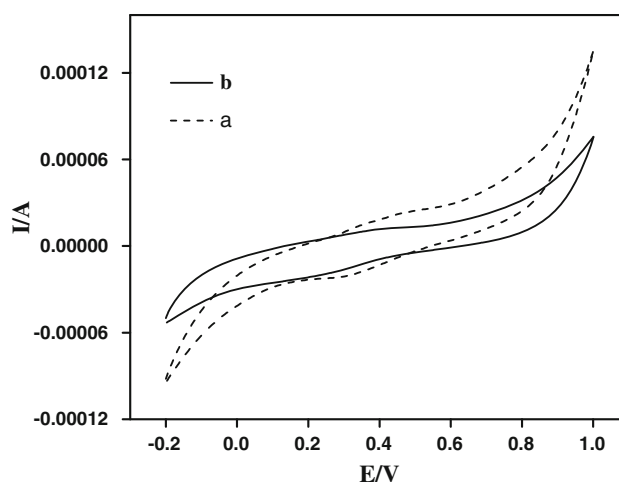


Fig. 5 Cyclic Voltammetry of chemically synthesized **a** self-doped poly(3-ABSA-co-AA), **b** PTSA poly(3-ABSA-co-AA)

for self-doped poly(3-ABSA-co-AA), and 0.46 V for PTSA-doped poly(3-ABSA-co-AA), whereas electrochemically synthesized polymer (Fig. 4) shows peaks at lower potential viz. 0.166 V for self-doped poly(AA) and 0.175 V for PTSA-doped poly(3-ABSA-co-AA). The shift in the peaks toward higher potential in the chemically synthesized samples can be attributed to the availability of less number of charge carriers (polarons).

Polymer films on a platinum electrode in PTSA medium exhibit electrochromic behavior on switching the potential between -0.2 and 0.8 V versus Ag/AgCl. These changes correspond to different oxidation states of all the samples. The electrochromic devices (ECDs) were constructed as shown in Fig. 6.

Figure 7a, b shows the electrochromic response of the polymer and copolymer films in 0.2 M PTSA medium. In the reduced state (-0.2 V) these films have a yellow color and become green at 0.3 V versus Ag/AgCl (Fig. 6). Increasing the potential above 0.4 V produces dark blue forms. The golden yellow to green transition is attributed to the oxidation of aromatic amine nitrogen to the stable radical cations; the second oxidation to dications induces a green to blue color transition. The difference in response time for oxidation and reduction can be explained by the different electrical conductivities of the two states. A life cycle test of the poly(AA) film in 0.2 M PTSA medium shows that on restricting the upper potential limit to $+0.4$ V, only 5% loss in the electrochemical activity of the polymer film is observed after 1×10^4 cycles, whereas for other investigated polymers it lies between 10 and 20%, for poly(3-ABSA-co-AA) again it is around 5%. No significant difference has been observed in the chronoamperometric response of poly(AA) and poly(3-ABSA-co-AA) (Fig. 7a, b) [41]. The rate of coloration is influenced by the ease of oxidation to its maximum state.

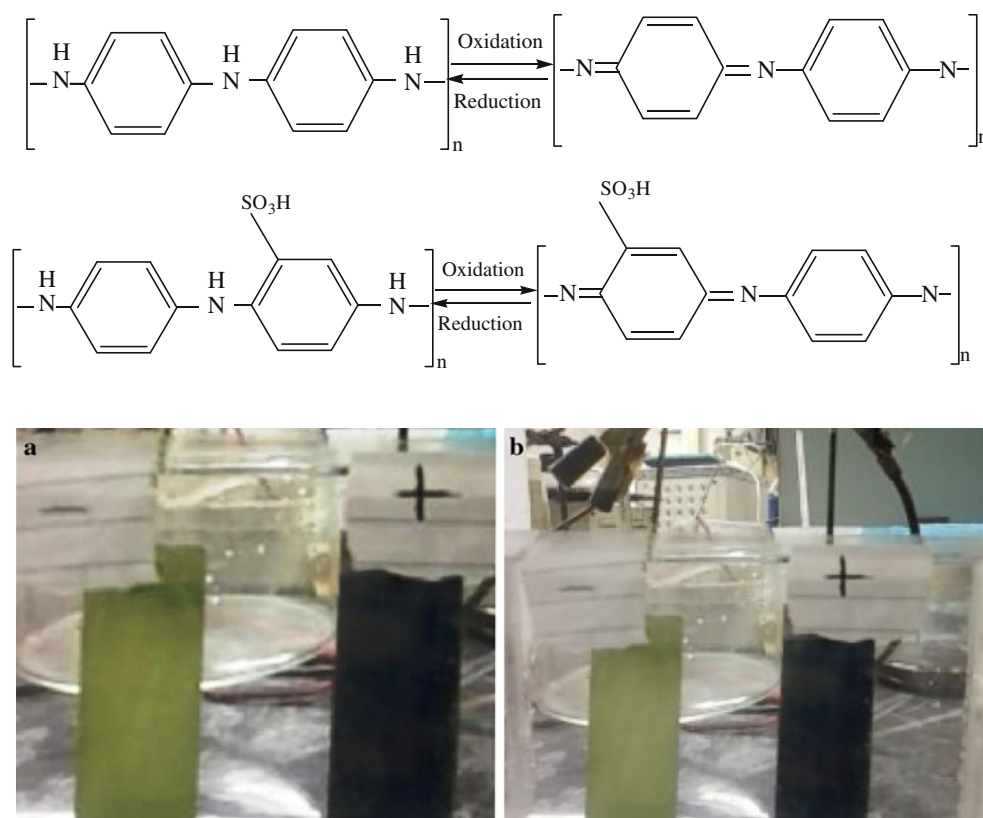


Fig. 6 Electrochromic device of **a** poly(AA) and **b** poly(3-ABSA-co-AA)

Although ABSA has improved the solubility of the polymers but it has no adverse effect on electrochromic response. For further improvement of electrochromic response, the copolymerization of poly(*o*-methoxy aromaticamine) (poly(3-ABSA-co-methoxy AA)) was carried out. It is evident from Fig. 7c that anodic peak current value increases by one order for (poly(3-ABSA-co-methoxy AA)); the attachment of a substituent on the aromatic ring shows a favorable effect on the electrochromic response time and improves life cycle. It was felt that apart from their chemical structure, the morphology may also play the important role in their switching performance. The SEM images of poly(AA), poly(3-ABSA-co-AA) and poly(3-ABSA-co-methoxy AA) are shown in Fig. 8. The poly(AA) shows a globular sponge-like structure (Fig. 8a), whereas, in the case of poly(3-ABSA-co-AA), hollow tube like morphology seems to be embedded in the globular pattern. ABSA as an intrinsic dopant may play an important role in achieving the microtubular morphology. In case of (poly(3-ABSA-co-methoxy AA)) the tubular morphology prevails all over the sample. This tubular morphology may be due to the arrangement of the polymer in 1D chain form, due to

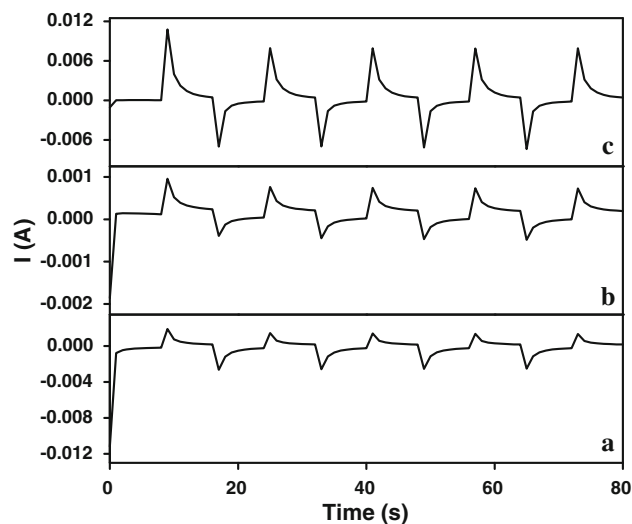


Fig. 7 Chronoamperometry of **a** poly(AA), **b** poly(3-ABSA-co-AA) and **c** poly(3-ABSA-co-methoxy-AA)

which the polymer may take less time for switching to same value of current or in the same time it may switch to higher values of current.

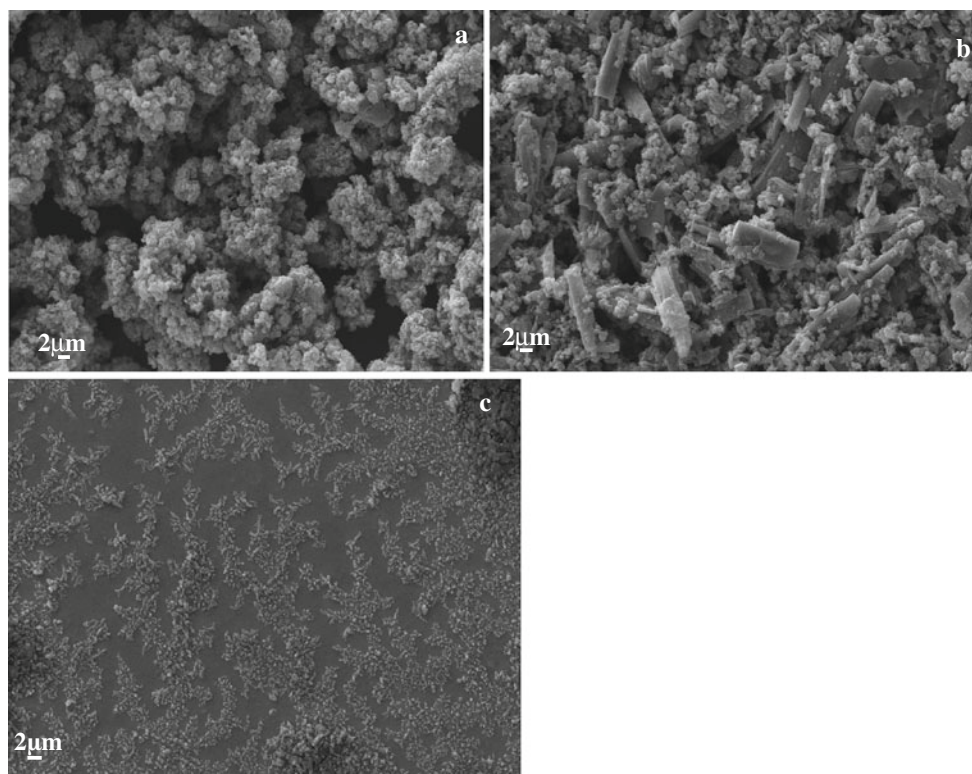


Fig. 8 SEM image of **a** poly(AA), **b** poly(3-ABSA-co-AA) and **c** poly(3ABSA-co-methoxy-AA)

4 Conclusions

The conductivity of homopolymers and copolymers is of the order of 10^{-4} to 10^{-2} S cm $^{-1}$ depending on the molar ratio of ABSA in the copolymer chain and the nature of the reaction media. The surface morphology of polyaromatic amines changes with introducing ABSA. The electrochromic switching ability of the devices was estimated from switching times between their oxidized and reduced states. It was observed that the copolymer poly(3-ABSA-co-methoxy AA) can be a promising candidate for electrochromic device.

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